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Absorption and Penetration Of Oil-soluble Wood Preservatives Applied by Dip Treatment

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THIS BULLETIN reports a portion of the results of a three-year research program on the absorption and penetration characteristics and relative effectiveness of a group of oil-soluble wood preservatives applied by simple treatment. The work was financially supported in part by the Chemicals and Plastics Division of the Quartermaster Research and Development Center, U. S. Army, Natick, Massachusetts. The research reported was completed under contracts DA-44-109-QM-1029 and DA-44-109-QM-1643 with effective dates June 7, 1952–June 6, 1953 and October 17, 1953–December 31, 1954 respectively.

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INTRODUCTION

Millions of cubic feet of wood products in various forms are treated each year with preservatives. These treatments greatly reduce decay and insect attack and thereby increase the useful life of wood. Wood used under conditions favoring severe deterioration is treated preferably by pressure processes Extensive service experience and field tests prove that pressure treatments with effective wood preservatives give excellent protection against biological deterioration. Pressure treatments, however, are not always feasible or necessary. Some factors which affect the choice of the preservative and type of treatment for a specific item are original costs, length of service life needed, necessity for cutting and shaping after treatment, conditions of exposure, and hazards involved during treatment and subsequent use of the treated material.

Treatment of wood with preservatives in penetrating solvents by dipping, soaking, and brushing is commonly known as simple treatment and has proven advantageous where the decay hazard is low or where protection is needed for a limited time. In the military services many occasions arise in the use of wood where only simple treatment methods are feasible.

Little basic information exists on the many factors affecting treatment of wood by simple methods. Data are needed on wood and method of treatment variables which affect absorption and penetration and on the period of protection to be expected from various preservative treatments exposed under different conditions of use. This research program represents only a partial solution of the many problems associated with simple treatments (their role in wood preservation and their relative effectiveness).

OBJECTIVES

The general purposes of this project were to study factors which affect the absorption and penetration of five oil-soluble preservatives applied by dipping, and to measure the relative effectiveness of these preservatives under a variety of test conditions. The specific purpose of the work reported in this bulletin was to obtain measurements of the effects of time of immersion and the specific gravity and moisture content of wood at treatment on the absorption and penetration of a group of oil-soluble preservatives applied by dipping.

LITERATURE REVIEW

Although many papers have been published on the general subject of dip treatment and other simple treatment methods (Hicock and Olson 1954; Hirt 1946; Verrall 1946) only a few are pertinent to this study. The effect of time of immersion on absorption and penetration has been evaluated, but the relation of specific gravity and moisture content has been almost completely disregarded. A limited literature exists on the methods used to determine the penetration of colorless preservatives.

Hubert (1938) tested the penetration of sugar pine, western white pine, and ponderosa pine sapwood by various solvents after a twenty-second dip treatment. He found depth of penetration to be greatest in sugar pine and least in ponderosa pine.

Buckman and Pera (1942) measured the effect of time and depth of immersion on the absorption and penetration of a kerosene solution of pentachlorophenol colored by an oil-soluble dye. The specific gravity of southern yellow pine sapwood specimens used in the study ranged from 0.41 to 0.48 (based on oven-dry weight and green volume) and moisture contents ranged from 10.9 to 15.6 per cent (based on oven-dry weight). Three different sizes of end-sealed test specimens were used. Absorption and penetration were determined for immersion periods of 0.25, 1.0, 4.0, and 24 hours. Nominal 1 x 6 inch material was completely penetrated in the 0.25-hour treatment.

In his work with balsa wood Rhoads (1948) tested the effect of specific gravity on the absorption of a pentachlorophenol solution applied by dip treatment. The density of the balsa specimens ranged from four to twenty-five pounds per cubic foot. Results were very variable, especially in the light- and medium-density wood. The higher specific gravity specimens were consistently low in absorption and penetration. In specimens with exposed end grain the principal pathway of absorption was through the vessels. In end-sealed specimens wood rays were chiefly responsible for absorption.

Schulze and Theden (1950) measured the absorption and penetration of "oily" wood preservatives as affected by moisture content and type of treatment. End-sealed pine sapwood specimens were used. In both brush and dip treatments they concluded that variation in moisture content of wood below the fiber saturation point had no effect on transverse absorption and penetration. They also concluded that moisture contents in wood above the fiber saturation point prevented penetration through transverse surfaces and greatly reduced absorption at end-grain surfaces. Penetration and absorption were found to be directly correlated, and transverse penetration was found to occur primarily along the wood rays.

Raphael and Graham (1951) tested the longitudinal penetration of several petroleum oils in coast type Douglas fir. Their specimens were end-dipped to a depth of four inches (not completely submerged) for fifteen minutes. Penetrations were measured eighteen hours after treatment and after an average lapse of thirty days. Test specimens were conditioned to twelve per cent moisture content and ranged in specific gravity from 0.470 to 0.625 (based on oven-dry weight and volume). No correlation was found between specific gravity and penetration, but this was qualified as follows: "...the small number of strips on which this information was based prevents the drawing of definite conclusions about the relationships of these variables [rings per inch, specific gravity, and per cent summerwood]."

Brown (1954) and Brown and Zabel (1954) stated that specific gravity and moisture content inversely affect the absorption of several oil-soluble preservatives by ponderosa pine sapwood.

Measurement of the penetration of colorless preservative solutions has been accomplished primarily with oil-soluble dyes. However, colorific methods based on reactions with toxic ingredients have been proposed for the determination of pentachlorophenol in treated wood (Chapman Chemical Co. 1952; Monsanto Chemical Co. 1955). The determination of copper naphthenate is specified by the American Wood Preservers' Association (1955). Also available for possible use are many organic indicators which give specific colorific reactions with metal constituents of preservative solutions (Feigl 1946).

MATERIALS AND METHODS

Experimental Design

A factorial design in twenty replications was used to measure absorption as affected by moisture content of wood at treatment (five levels), specific gravity of wood (three ranges), and preservatives (five solutions). Absorptions were measured at stated intervals through a ten-minute immersion period. Further details of the various levels of the main factors are discussed later.

After the absorption measurements were completed, ten specimens were randomly selected from each subgroup of twenty for installation in an outdoor stake test. From the remaining ten specimens of each subgroup, five were selected for determination of penetration resulting from the ten-minute immersion period. Additional specimens were treated to permit penetration measurements from fifteen-second and three-minute immersion periods. The last five specimens were retained for possible future use.

This bulletin reports only the results of absorption and penetration determinations.

Absorption Studies

Preparation of test specimens.—Test specimens were prepared from ponderosa pine (*Pinus ponderosa* Laws.) purchased from local lumberyards. The specimens were sapwood, free of stain and other defects, with a slope of grain less than one inch per twenty inches. Specimens were 0.75 inch on the radial face, 0.50 inch on the tangential face, and 6.0 inches long.

Grouping of test specimens.—The specimens used in these studies were prepared in two separate groups, but for the sake of simplicity their grouping will be described as if it were accomplished in one operation. All specimens were oven dried in a forced draft oven at 105°C. Specific gravities were computed based on the oven-dry weight and volume.

The required total population of specimens was chosen from an excess number and divided into three approximately equal groups with the following specific gravity ranges: A. 0.33-0.39: B, 0.40-0.44: C. 0.45-0.61. From each specific gravity range twenty-five subgroups of twenty specimens each were chosen randomly. Each of the resulting seventy-five subgroups represented a specific gravity range to be conditioned to a particular moisture content for treatment with a specific preservative.

All specimens were labelled as to specific gravity range. moisture content level, preservative solution. actual specific gravity, and subgroup sample number.

Conditioning to desired moisture contents.—The effect of moisture content was studied at the oven-dry condition and at twelve, twenty-five. thirty-five. and fifty per cent (based on oven-dry weight).

Specimens to be treated at the oven-dry condition were dried at 105°C. for twenty-four hours, removed from the oven individually. placed in a desiccator containing phosphorus pentoxide. and allowed to cool to room temperature before treatment.

Specimens to be treated at twelve per cent moisture content were conditioned to this level in a constant humidity room. Immediately prior to treatment small groups of specimens were placed in a desiccator containing a saturated aqueous solution of potassium nitrate and ammonium chloride.[1] The specimens were treated immediately on removal from the container.

Specimens to be treated at twenty-five per cent moisture content were first conditioned to twelve per cent in the constant humidity room. They were then dipped in distilled water for a length of time predetermined as adequate to increase the moisture content to approximately twenty-five per cent. The specimens were then stored in desiccators over a free water surface at 3°C. for at least one month prior to treatment. The specimens were allowed to come to room temperature before treatment.

Specimens to be treated at thirty-five and fifty per cent moisture content were conditioned similarly to the twenty-five per cent group. Wet weights for the desired moisture contents were calculated for individual specimens from known oven-dry weights. and specimens were soaked in distilled water until the calculated wet weights had been attained. Again specimens were stored in desiccators at 3°C. for approximately five weeks prior to treatment and were allowed to come to room temperature before treatment.

Tests were conducted on samples from the various moisture content levels to determine moisture gradients within the specimens at the time of treatment. No measurable gradients were present at the oven-dry and twelve per cent moisture contents. At the other levels maximum differences from end to

[1] At 30°C. this solution maintained a relative humidity adequate for maintaining wood at approximately twelve per cent moisture content.

TABLE 1

Toxicant concentrations and certain physical characteristics for the five preservative solutions included in the study are presented. All solutions were prepared with Solvasol No. 5 as the solvent and contained a uniform water repellent combination of paraffin and Pentalyn H (Appendix I). All values are means of three samples.

Concentration of Toxicant in per cent[a]	Relative Viscosity[b]	Surface Tension[c] dynes per cm.	Specific Gravity[d]
5 per cent pentachlorophenol	1.586	24.8	0.799
2 per cent metallic copper (as copper naphthenate)	3.279	25.0	0.824
1.1 per cent copper-8-quinolinolate	1.556	24.7	0.786
7.5 per cent rosin amine D pentachlorophenate	1.955	24.9	0.808
5 per cent zinc alkyl sulphonate	1.775	24.4	0.789

[a] All per cent concentrations are calculated on a weight basis.
[b] Relative viscosities determined by Ostwald viscosimeter No. 50 at temperature of 30°C. Relative viscosities are based on Solvasol No. 5 as 1.000.
[c] Determined using a Cenco-du Nouy Tensiometer with solution at a temperature of 23°C.
[d] Specific gravities based on solution volume and weight at 20°C.

center of the specimen did not exceed five per cent and between shell and core at the center of the specimen did not exceed two per cent. The greatest gradients occurred at the fifty per cent moisture content.

Preservative solutions.—Major producers were requested to recommend the most widely used or promising wood preservatives for inclusion in the study. The preservatives selected (Table 1) were those of greatest interest to the Quartermaster Corps. Although some work was done with proprietary compounds the results are restricted to preservative solutions prepared by the authors. The solutions were prepared using Solvasol No. 5[2] as the diluent and a water repellent system composed of refined paraffin (Fisher NF) and Pentalyn H,[3] a resin produced by the Hercules Powder Company. The composition of the preservative solutions is given in Appendix I. The preservatives tested were five per cent pentachlorophenol, two per cent metallic copper (as copper naphthenate), 1.1 per cent copper-8-quinolinolate, 7.5 per cent rosin amine D pentachlorophenate, and five per cent zinc alkyl sulphonate.

Measurement of absorption.—A method was developed which permitted the measurement of absorption by a single specimen at various time intervals; the specimen remained submerged throughout these measurements. The specimen was placed in a weighted jig and suspended in the preservative solution from the left end of a triple beam balance (Fig. 1). The submerged weight of

[2] Solvasol No. 5 is a Socony-Vacuum Oil Company, Inc., product with approximately 20 per cent aromatic content and a flash point of 103°F.

[3] Pentalyn H is the pentaerythritol ester of hydrogenated rosin.

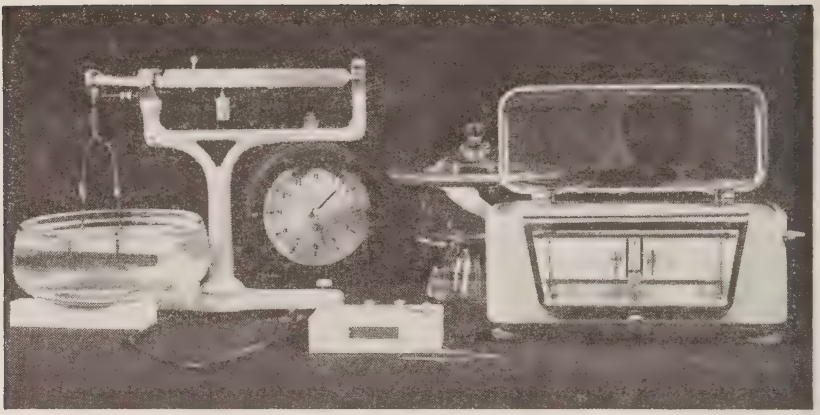

FIG. 1.—Apparatus used for measuring the absorption of the preservative solutions by test specimens. (*Photograph by Wilfred A. Côté, Jr.*)

the specimen and jig was determined to the nearest centigram at fifteen and thirty seconds after immersion and thereafter at thirty-second intervals through a ten-minute dip period. Since the volume of the specimen remained constant, the change in submerged weight was a measure of absorption. Total absorption was the difference between the post-treatment weight (after ten minutes of immersion) and the pre-treatment equilibrium weight. The initial absorption was that which occurred during the first 0.25 minutes of immersion. It was the difference between total absorption and the absorptions occurring during the 0.25-10.0 immersion period. The latter were measured successively by changes in balance readings.

PENETRATION STUDIES

The penetration of the preservatives was determined from test specimens immersed for fifteen seconds, three minutes, and ten minutes. Penetration data were obtained for the three wood planes: longitudinal, radial, and tangential.

Chemical indicators.—All penetrations were determined by the use of chemical indicators which gave colorific reactions with the preservative or a portion of the preservative being studied.

A 1.0 per cent solution of dithio-oxamide in ethyl alcohol was used as the indicator for copper naphthenate and copper-8-quinolinolate. This indicator formed a black or bluish-black precipitate upon contact with a copper-containing preservative and remained an indefinite period. The indicator was applied by lightly spraying the wood surfaces.

A 0.006 per cent solution of diphenyl-thio-carbazone in carbon tetrachloride was used as the indicator for the preservative containing zinc alkyl sulphonate. A deep red color developed on contact of the indicator and the

TABLE 2

Approximate minimum concentrations of toxicants in treating solutions for which the chemical indicators gave a positive colorific reaction in dip-treated ponderosa pine sapwood.

Toxicant	Indicator	Concentration (per cent of toxicant in treating solution)
Pentachlorophenol	N,N-dimethylaniline and petroleum ether (equal parts by volume)	0.25
Metallic copper (as copper naphthenate)	1 per cent dithio-oxamide in ethyl alcohol	0.005
Copper-8-quinolinolate	1 per cent dithio-oxamide in ethyl alcohol	0.10
Rosin amine D pentachlorophenate	N,N-dimethylaniline and petroleum ether (equal parts by volume)	0.19
Zinc alkyl sulphonate	0.006 per cent diphenylthio-carbazone in carbon tetrachloride	0.12

zinc-containing preservative. The color gradually faded within a few days of treatment. The indicator was applied by lightly spraying the wood surfaces.

Penetrations of pentachlorophenol and rosin amine D pentachlorophenate were determined by exposing the test specimens to chlorine dioxide gas and then spraying them with a mixture (equal parts by volume) of petroleum ether and N,N-dimethylaniline. This procedure has been published by the Chapman Chemical Company (1952).[4] Test specimens were exposed to chlorine dioxide for approximately one hour and sprayed immediately with the indicator. Penetration of the preservative was shown by a violet color. Measurements of penetration were made rapidly since unpenetrated wood became colored after approximately ten minutes.

The approximate minimum concentrations of toxicants in treating solutions for which the chemical indicators give colorific reactions in dip-treated wood are listed in Table 2. Close agreements were obtained when the colorific chemical indicators were compared with oil-soluble dyes on freshly treated specimens.

Test specimens. -Penetration data after a ten-minute immersion were obtained from the specimens used in the absorption studies. These data were obtained at all the levels of specific gravity and moisture content previously designated. After the ten-minute dip treatment, specimens were loosely piled

[4] Essentially the same procedure was published in Germany (Sandermann and Jonas 1951) and recently modified in this country by the Monsanto Chemical Company (1955). The recent modification used a more sensitive indicator and results indicated that more precise color zones could be obtained in a nitrogen atmosphere.

until weight losses indicated that the preservative solvent had evaporated. Specimens were then bulk-piled under conditions which maintained the wood below a ten per cent moisture content. The storage period between treatment and penetration determinations ranged from two to nine months.

Penetration data for the fifteen-second and three-minute immersion periods were obtained from additional specimens treated specifically for this purpose. The data were obtained for all levels of specific gravity but were restricted to the twelve per cent moisture content level. After treatment the specimens were loosely piled until the solvent had evaporated and then used immediately for penetration determinations.

Penetration measurements were made on five specimens in each treatment.

Preparation of specimens for penetration determinations.—Each specimen was sawed transversely into halves, and each half was appropriately labelled. One of the halves was split so as to expose radial surfaces, and the other was split so as to expose tangential surfaces. The splitting was done from freshly sawed surfaces and in a manner such that the splitting plane was through the approximate center of each half.

Penetration measurements.—All measurements were made immediately after the application of the chemical indicator. Penetrations were measured to the nearest millimeter for each wood plane and were averaged for the planes exposed per specimen. Longitudinal penetrations were determined on radial surfaces and did not consider occasional streaks of penetration limited to summerwood areas of an annual ring.

RESULTS

Absorption Studies

A selected portion of the absorption data is presented in Table 3. These data are the mean absorption values obtained for each subgroup at three time intervals: fifteen seconds. three minutes, and ten minutes. The three-minute and ten-minute time intervals were chosen because they bracketed the range of times commonly used in dip treatments. The fifteen-second time interval was chosen because a large proportion of the absorption occurred in this short interval of immersion.

Analyses of variance of the total body of absorption data for each of the three time intervals is shown in Table 4. Standard methods of analysis as outlined by Cochran and Cox (1950) for factorial experiments were used.

Statistically. the effects of preservatives were significantly different; however. in view of the comparatively large variance ratios for moisture content and specific gravity it is doubtful that the variation among preservatives has any practical significance. For this reason the average absorption of all preservative solutions is shown in Figure 2. This series of curves shows the effects of time of immersion and specific gravity and moisture content of the wood on absorption.[5]

The major differences among mean absorption data were caused by differences in time of immersion and moisture content and specific gravity of the wood. The absorption of the preservative solutions. as expected, increased with time of immersion (Fig. 2). The initial absorption[6] was approximately

[5] Within each subgroup of twenty specimens, those specimens which exhibited a total absorption outside the range determined by the mean plus or minus 2.5 standard deviations were excluded from all further calculations. Twenty specimens of the 1500 total were so excluded and retained for subsequent observation.

[6] "Initial absorption" is defined as that absorption which occurred during the first fifteen seconds of immersion.

TABLE 3

Mean absorption of preservative solutions by test specimens of ponderosa pine sapwood. The data are classified by specific gravity, moisture content, and time of immersion. Specific gravity ranges were as follows: A (0.33–0.39), B (0.40–0.44), and C (0.45–0.61).

Preservatives	Time of Immersion (minutes)	Preservative Absorption (grams) at Various Moisture Contents (Per Cent)															Row Means	Preservative Means
		Oven-dry			12			25			35			50				
		Specific Gravity			Specific Gravity			Specific Gravity			Specific Gravity			Specific Gravity				
		A	B	C	A	B	C	A	B	C	A	B	C	A	B	C		
5 per cent pentachlorophenol	0.25	1.02	.87	.79	.96	.88	.77	.99	.91	.88	.51	.54	.34	.31	.32	.24	.69	
	3.00	1.50	1.27	1.18	1.30	1.10	1.00	1.13	1.05	1.01	.55	.63	.38	.34	.34	.26	.87	
	10.00	1.84	1.57	1.48	1.53	1.28	1.17	1.26	1.26	1.15	.60	.73	.43	.37	.37	.27	1.02	.86
2 per cent metallic copper (copper naphthenate)	0.25	1.00	.96	.80	.98	.92	.81	1.03	.89	.91	.52	.54	.36	.41	.40	.22	.72	
	3.00	1.46	1.38	1.29	1.27	1.19	1.00	1.13	.97	.98	.57	.60	.39	.44	.41	.22	.89	
	10.00	1.81	1.70	1.68	1.48	1.49	1.19	1.33	1.12	1.07	.61	.67	.41	.48	.43	.20	1.04	.88
1.1 per cent copper-8-quinolinolate	0.25	1.05	.94	.85	.94	.89	.82	.89	.76	.77	.49	.50	.40	.31	.40	.21	.68	
	3.00	1.63	1.36	1.29	1.21	1.07	1.02	1.00	.89	.87	.52	.55	.43	.33	.44	.21	.86	
	10.00	2.02	1.66	1.63	1.45	1.21	1.15	1.13	1.03	1.01	.56	.63	.47	.34	.46	.19	1.00	.85
7.5 per cent rosin amine D pentachlorophenate	0.25	1.12	.98	.91	.98	.92	.81	1.07	.82	.91	.50	.46	.30	.33	.35	.17	.71	
	3.00	1.70	1.43	1.36	1.20	1.15	1.00	1.17	.91	1.00	.53	.53	.32	.35	.37	.18	.88	
	10.00	2.02	1.73	1.65	1.34	1.31	1.15	1.29	.99	1.15	.56	.61	.34	.39	.42	.18	1.01	.87
5 per cent zinc alkyl sulphonate	0.25	1.05	.94	.95	.97	.94	.84	.88	.82	.82	.49	.43	.31	.30	.21	.15	.67	
	3.00	1.54	1.36	1.38	1.23	1.17	1.06	.98	.98	.89	.54	.48	.33	.33	.24	.16	.84	
	10.00	1.83	1.64	1.67	1.41	1.37	1.23	1.09	1.15	.98	.61	.55	.36	.36	.27	.14	.98	.83
Column Means		1.50	1.32	1.26	1.22	1.13	1.00	1.09	.97	.96	.54	.56	.37	.36	.36	.20		
Moisture Content Means		1.36			1.12			1.01			.49			.31				

TABLE 4

Analyses of variance of the absorption of five oil-soluble wood preservatives by laboratory specimens of ponderosa pine sapwood as affected by moisture content and specific gravity of the wood. Separate analyses were computed for fifteen-second, three-minute, and ten-minute immersion periods.[a]

Source of Variation	Degrees of Freedom	Fifteen-second Immersion		Three-minute Immersion		Ten-minute Immersion	
		Mean Square	Variance Ratio[b]	Mean Square	Variance Ratio[b]	Mean Square	Variance Ratio[b]
Preservatives	4	.0957	6.79**	.0817	2.98**	.1510	3.63**
Moisture content ...	1	27.4504	1946.84**	61.8412	2256.98**	96.1343	2310.92**
Specific gravity ..	2	2.7428	194.52**	4.5536	166.19**	5.9442	142.89**
P x MC[c]	16	.0999	7.09**	.1435	5.24**	.1625	3.91**
P x SG.....	8	.0389	2.76**	.0258	.94	.0291	.70
MC x SG....	8	.1292	9.16**	.2572	9.39**	.4219	10.14**
P x MC x SG	32	.0198	1.40	.0474	1.73**	.0862	2.07**
Within subgroups (error).....	1405	.0141	.....	.0274	.....	.0416	.....
Total	1479	.....	.....	.....	.....	.....	.....

** Significant at the 1 per cent level.

[a] Moisture content levels were: oven-dry condition and 12, 25, 35 and 50 per cent based on oven-dry weight. Specific gravity levels included 3 ranges: 0.33–0.39, 0.40–0.44, and 0.45–0.61.

[b] All variance ratios were obtained by using the mean square for "within subgroups" (1405 degrees of freedom) as the denominator of the variance fraction.

[c] Abbreviations are as follows: P—preservative, MC—moisture content, and SG—specific gravity.

half the total absorption. After this initial absorption the rate of absorption decreased with time of immersion. Mean absorption values for the three time intervals were as follows:

Time of immersion in minutes	*Mean absorption in grams*
0.25	0.69
3.00	0.87
10.00	1.01

An increase in moisture content of the test specimens affected both total and initial absorption. At the oven-dry condition approximately half of the total absorption occurred within the fifteen-second measurement, and as the moisture content increased this initial absorption accounted for an increasingly higher proportion of the total. For specimens above the fiber saturation point the initial absorptions were, effectively, the total absorptions. In addition, increased moisture content caused a decrease in the amount of initial

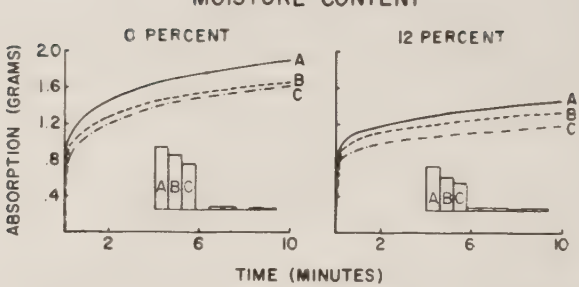

FIG. 2.—The mean absorption, averaged across the five preservative solutions, by ponderosa pine sapwood as affected by time of immersion and specific gravity and moisture content of the wood. The specific gravity ranges were as follows: A (0.33-0.39), B (0.40-0.44), C (0.45-0.61). Bar graphs are mean penetration for longitudinal, radial, and tangential directions (left to right) by specific gravity ranges.

absorption. At the oven-dry condition, twelve per cent and twenty-five per cent moisture contents, initial absorptions were approximately the same; however, at thirty-five and fifty per cent the initial absorptions were greatly reduced.

Absorption varied inversely with specific gravity, and mean absorption values for the three specific gravity ranges were as follows: A. 0.94 grams; B. 0.87 grams; C. 0.76 grams. An increase in moisture content of the test specimens caused a slight change in the effect of specific gravity. In order to demonstrate these effects clearly the data were regrouped by individual specific gravity values and moisture contents for the fifteen-second, three minute, and ten-minute immersion periods. This regrouping was done across preservatives on the assumption that no important differences existed among preservatives. Curves showing the regression of total absorption on specific gravity were balanced by the method of least squares. These curves were grouped by immersion times and moisture contents and are shown in Figure 3. Corresponding values for "a" (y-intercept) and "b" (slope of line) are shown in Table 5.

The slope of each line is a measure of the effect of specific gravity on absorption for that particular moisture content and time of immersion. The effect is an average over all the preservatives. Slope of line values ("b") demonstrate a considerable reduction between the twelve and twenty-five per cent moisture content levels (Table 5). This indicates that specific gravity has less influence on absorption at the three higher moisture contents than at the two lower moisture contents.

The analyses of variance (Table 4) supported the above results. The very high variance ratios for moisture content and specific gravity indicated that these two variables were of outstanding importance. The third highest variance ratio was obtained for the moisture content by specific gravity interaction. This also supported a previous observation; namely, that the effect of specific

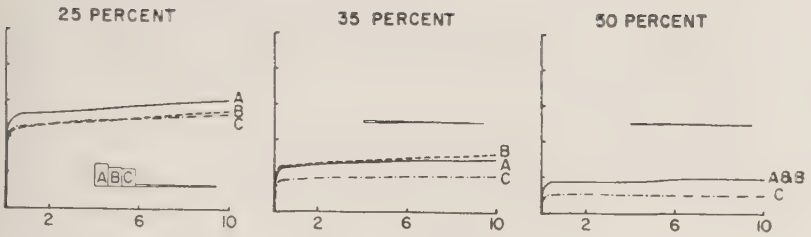

FIG. 2 — (*Continued*)

gravity on absorption varied with changes in moisture content. The effects of preservatives, as well as other interactions, were also highly significant. However, in view of the magnitude of the moisture content and specific gravity variance ratios, it was doubtful that any other source of variation tested (though highly significant statistically) would have any practical importance.

The mean absorption data (Table 3 and Fig. 2) indicated a sharp reduction from the twenty-five to the thirty-five per cent moisture content level. It therefore seemed desirable to determine what proportion of the variance due to moisture content and its interactions could be attributed to a grouping of the three lower levels (oven-dry, twelve per cent, and twenty-five per cent) and the two higher levels (thirty-five and fifty per cent).[7] Accordingly, an

[7] It was assumed that the fiber saturation point of ponderosa pine was between twenty-five and thirty-five per cent moisture content.

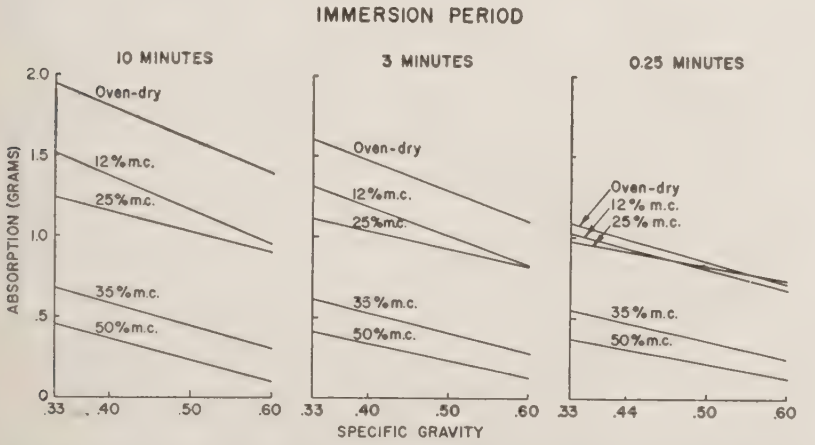

FIG. 3.—The regression of preservative solution absorption on specific gravity of test specimens as determined for each moisture content level at fifteen-second, three-minute, and ten-minute immersion period. Each line is the average over all preservative solutions.

TABLE 5

Tabulation of "a" (y-intercept) and "b" (slope) for the regression lines plotted in Figure 3.

Equation Constants	Time of Immersion in Minutes	Moisture Content in Per Cent				
		0	12	25	35	50
a	10	2.61	2.20	1.67	1.13	0.88
	3	2.24	1.92	1.49	1.02	0.75
	0.25	1.54	1.45	1.28	0.92	0.68
b	10	—2.04	—2.08	—1.28	—1.38	—1.29
	3	—1.92	—1.84	—1.13	—1.23	—1.03
	0.25	—1.37	—1.29	—0.90	—1.12	—0.93

TABLE 6

Analysis of variance of fifteen-second absorption data with "sums of squares" for moisture content, moisture content X specific gravity interaction, and preservative X moisture content interaction divided so as to permit isolation of the variance due to a comparison between bodies of data obtained above and below the fiber saturation point.[a]

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Square	Variance Ratio
Preservatives	4	0.3827	0.0957	6.79**
Moisture content				
above f.s.p. vs. below f.s.p.	1	105.5088	105.5088	7482.89**
among all levels	3	4.2928	1.4309	101.48**
Specific gravity	2	5.4856	2.7428	194.52**
Preservatives x moisture content				
preservatives x above f.s.p. vs. below f.s.p.	4	1.4707	0.3676	26.07**
preservatives x among all levels	12	0.1278	0.0106	0.75
Preservatives x specific gravity	8	0.3114	0.0389	2.76**
Moisture content x specific gravity				
specific gravity x above f.s.p. vs. below f.s.p.	2	0.5905	0.2952	20.93**
specific gravity x among all levels	6	0.4434	0.0739	5.24**
Preservatives x moisture content x specific gravity	32	0.6330	0.0198	1.40
Within subgroups	1405	19.8141	0.0141	
Total	1479			

** Significant at the 1 per cent level.

[a] Moisture content levels were: oven-dry and 12, 25, 35 and 50 per cent based on oven-dry weight. The fiber saturation point (f.s.p.) was assumed to fall between the 25 and 35 per cent levels.

analysis was made of the fifteen-second absorption data to permit this comparison.[8] The results of this analysis are shown in Table 6.

In each of the variances divided it was shown that a large proportion of the variance reported in Table 4 could be attributed to a comparison between bodies of data obtained above and below the fiber saturation point. However, it was shown that in the cases of the moisture content main effect and moisture content by specific gravity interaction, significant differences still existed, either among the three lower moisture content levels or between the twenty-five and thirty-five per cent levels.

Since so much variation was shown to exist between the bodies of data obtained above and below the fiber saturation point an additional analysis was made using fifteen-second absorption data from the three lower moisture content levels only. This analysis is presented in Table 7. Again, the magnitude of the variance ratios for moisture content and specific gravity indicated that these two variables were of the greatest importance.

Effect of Method of Drying on Absorption

The possibility that oven-drying for the determination of specific gravity values effected preservative solution absorption motivated a study of this factor. A factorial experiment in seventeen replications was designed to measure the effect of initial drying temperature (three levels) and moisture content at time of treatment (three levels) on absorption.

[8] The procedure used followed the method outlined by Snedecor (1946) pp. 278-280.

TABLE 7

Analysis of variance of fifteen-second absorption data for oven-dry, twelve per cent and twenty-five per cent moisture content levels.

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Square	Variance Ratio
Preservatives	4	0.4698	0.1174	6.24**
Moisture content	2	0.6186	0.3093	16.45**
Specific gravity	2	3.3781	1.6890	89.84**
Preservatives x moisture content	8	0.8471	0.1059	5.63**
Preservatives x specific gravity	8	0.2456	0.0307	1.63
Moisture content x specific gravity	4	0.0994	0.0248	1.32
Preservatives x moisture content x specific gravity	16	1.5713	0.0982	5.22**
Within subgroups	840	15.8219	0.0188	
Total	884	23.0518		

** Significant at the 1 per cent level.

Test specimens of the same dimensions and grade as those used in the main absorption study were prepared from a freshly-felled red pine (*Pinus resinosa* Ait.) approximately twenty inches in diameter at breast height.

The green specimens were randomly divided into three groups (fifty-one specimens per group) with each group to be dried under different conditions. Group 1 specimens were dried in an oven at 104°C. to the oven-dry condition. Group 2 specimens were dried to four per cent moisture content under conditions simulating kiln drying. Group 3 specimens were air dried to ten per cent moisture content.

After drying, each group was further subdivided into three subgroups (designated A, B, and C) of seventeen specimens each. Subgroups A were conditioned to approximately 0.5 per cent moisture content in a desiccator containing phosphorus pentoxide. Subgroups B were conditioned to twelve per cent moisture content in a constant temperature-humidity room. Subgroups C were soaked in distilled water until they had reached approximately twenty-five per cent moisture content and then stored in a desiccator over a free water surface at 3°C. for six weeks prior to treatment.

All specimens were dip treated for six minutes in a solution containing two per cent metallic copper (as copper naphthenate). Specimens were weighed to the nearest centigram immediately before and after treatment. Total preservative absorption was used as the criterion for comparison.

The results of this experiment are summarized in Table 8. These data present mean total absorption by seventeen specimens per subgroup as affected by drying method and moisture content at time of treatment.

Mean total absorption varied directly with drying temperature and inversely with moisture content at time of treatment. Absorption by specimens

TABLE 8

Mean absorption of a two per cent metallic copper (as copper naphthenate) solution by red pine specimens dried by three different methods and dip treated for six minutes at three different moisture content levels. Drying methods included oven drying, kiln drying, and air drying, and treatment moisture contents included oven-dry, twelve per cent, and twenty-five per cent. Each value is the mean of data from seventeen specimens.

Treatment Moisture Content (per cent)	Mean Absorption (grams)			Mean
	Method of drying			
	Oven-dried	Kiln-dried	Air-dried	
Oven-dry	1.544	1.444	1.396	1.461
12	1.486	1.335	1.288	1.370
25	1.332	1.239	1.189	1.253
Mean	1.454	1.339	1.291	1.361

TABLE 9

Analysis of variance of absorption data as affected by the method of drying specimens and moisture content at time of treatment.

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Square	Variance Ratio
Method of drying	2	0.7113	0.3557	27.15**
Treatment moisture content	2	1.1091	0.5546	42.34**
Interaction	4	0.0218	0.0055	0.42
Within subgroups	144	1.8796	0.0131	
Total	152	3.7218		

** Significant at the 1 per cent level.

which had been oven-dried was approximately twelve per cent greater than absorption by air-dried specimens and was intermediate for kiln-dried specimens. Absorption by specimens treated at the oven-dry moisture content, regardless of drying temperature, was approximately seventeen per cent greater than that of specimens at twenty-five per cent moisture content.

An analysis of variance of the data is shown in Table 9. The effects of both drying temperature and moisture content were highly significant, thereby supporting the observations noted above. The interaction of moisture content by drying temperature was shown to be non-significant. Although the drying temperature had a marked effect on absorption, this effect remained constant throughout the experiment. The effect of moisture content on absorption was not changed with changes in drying procedure.

Penetration Studies

Average longitudinal, radial, and tangential penetrations obtained in a ten-minute dip treatment for the five preservative solutions are presented in Table 10. Graphical representations of the average longitudinal penetration for all preservatives are presented in Figure 2. Penetrations obtained from fifteen-second and three-minute treatments are presented in Table 11. Ten-minute data were obtained at all levels of specific gravity and moisture content, but fifteen-second and three-minute data were restricted to the twelve per cent moisture content level.

The data in Tables 10 and 11 indicated the following:

Almost no penetration was obtained in specimens treated at the thirty-five and fifty per cent moisture content levels.

Penetration of specimens dipped for fifteen seconds was more than half that of specimens immersed for ten minutes.

Among the three lower moisture contents, radial and tangential penetrations were very uniform.

TABLE 10

Penetration of oil-soluble preservatives obtained in ponderosa pine sapwood by a ten-minute dip treatment. Determinations were made with colorific indicators after the preservative solvent had evaporated. Specific gravity ranges were as follows: A (0.33–0.39), B (0.40–0.44), C (0.45–0.61). Each figure is the mean of data from five specimens.

Preservatives	Direction of Penetration	Mean Penetration (millimeters) at Various Moisture Contents (Per Cent)														
		Oven-dry			12			25			35			50		
		Specific Gravity			Specific Gravity			Specific Gravity			Specific Gravity			Specific Gravity		
		A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
5 per cent pentachlorophenol	Long.	75	64	54	57	40	31	35	28	27	2	2	2	1	1	1
	Rad.	..[a]	4	4	3	3	3	3	3	3	1	1	T[b]	T	T	T
	Tang.	..	2	1	1	1	1	1	1	1	T	T	T	T	T	0
2 per cent metallic copper (copper naphthenate)	Long.	65	55	41	51	39	31	35	29	28	4	4	2	2	3	1
	Rad.	3	4	4	4	3	3	3	3	3	1	2	1	1	1	T
	Tang.	2	2	2	2	2	2	2	2	1	1	1	T	T	T	0
1.1 per cent copper-8-quinolinolate	Long.	72	63	51	53	44	30	16	15	16	2	1	1	1	1	1
	Rad.	..	3	3	2	2	3	2	2	2	1	1	T	1	T	1
	Tang.	..	2	2	1	2	2	1	1	1	T	T	T	T	T	T
7.5 per cent rosin amine D pentachlorophenate	Long.	75	69	58	44	25	28	27	21	22	2	2	1	2	1	T
	Rad.	..	3	3	3	3	3	2	1	1	1	T	T	T	T	T
	Tang.	..	1	1	1	1	1	1	0	0	T	T	T	T	T	0
5 per cent zinc alkyl sulphonate	Long.	60	51	51	37	32	28	11	11	8	1	T	T	1	T	0
	Rad.	3	3	3	2	3	3	2	2	1	1	T	T	1	T	0
	Tang.	2	2	2	1	1	1	1	1	0	1	T	T	T	T	0

[a] Complete longitudinal penetration masked side penetration.

[b] T indicates very slight penetration (trace).

TABLE 11

Penetration of oil-soluble preservatives obtained in ponderosa pine sapwood by fifteen-second, three-minute, and ten-minute dip treatments. Test specimens were conditioned to twelve per cent equilibrium moisture content before treatment. Penetrations were determined with colorific indicators after the preservative solvent had evaporated. Specific gravity ranges were as follows: A (0.33-0.39), B (0.40-0.44), C (0.45-0.61). Each figure is the mean of data from five specimens.

Preservatives	Direction of Penetration	Mean Penetration (millimeters) at Various Times of Immersion								
		0.25 minutes			3 minutes			10 minutes		
		Specific Gravity			Specific Gravity			Specific Gravity		
		A	B	C	A	B	C	A	B	C
5 per cent pentachlorophenol	Long.	45	35	26	47	43	29	57	40	31
	Rad.	4	3	3	3	3	3	3	3	3
	Tang.	1	1	1	1	1	1	1	1	1
2 per cent metallic copper (copper naphthenate)	Long.	34	29	26	37	31	29	51	39	31
	Rad.	3	3	3	2	2	3	4	3	3
	Tang.	1	1	1	1	1	1	2	2	2
1.1 per cent copper-8-quinolinolate	Long.	39	34	27	45	32	30	53	44	30
	Rad.	2	2	2	3	3	3	2	2	2
	Tang.	1	1	1	1	1	1	1	1	1
7.5 per cent rosin amine D pentachlorophenate	Long.	39	30	28	38	38	23	44	25	28
	Rad.	3	3	3	2	2	3	3	3	3
	Tang.	1	1	1	1	1	1	1	2	2
5 per cent zinc alkyl sulphonate	Long.	25	19	18	27	18	16	37	32	28
	Rad.	2	2	2	2	2	2	2	3	3
	Tang.	1	1	1	1	1	1	1	1	1

The ten-minute longitudinal penetration data varied inversely with specific gravity and moisture content.

A comparison of longitudinal penetration and ten-minute absorption data (Table 3) suggested a possible correlation between these values. Accordingly a correlation coefficient was computed using 218 paired values of absorption and penetration from the three lower moisture content levels. The resulting correlation coefficient ($r = 0.752$) indicated that penetration was closely related to absorption and was similarly affected by specific gravity and moisture content of the wood. At the twenty-five per cent moisture content the penetration of zinc alkyl sulphonate and copper-8-quinolinolate was considerably less than with the other solutions.

Certain generalizations about the penetrations of the various solutions into the test specimens can be made. The deepest penetrations were longitudinal and the least were tangential. A deeper penetration occurred in the summerwood than in the spring wood portion of an annual ring. The penetration patterns of various solutions were similar with the exception of zinc alkyl sulphonate where penetration was largely limited to the summerwood. Considerable penetration along the radial planes occurs in the wood rays. Occasional splits on the tangential planes exposing resin canals indicate deep penetrations in these tissues. Occasional samples within groups where factors under study were under control demonstrated penetration ranging from essentially none to complete.

DISCUSSION

The absorption of preservative solutions by wood is a complex phenomenon involving many variables. This study of the effects of certain factors on absorption and penetration was limited to dip treatments of ponderosa pine sapwood using a single solvent and water repellent system. The data are suggestive for other solvent systems and wood species, but extrapolation to other conditions without reservation would be questionable. The test specimens were oven-dried for specific gravity determinations and this was shown to increase total absorption; however, the effect of moisture content on absorption was not altered. The absorption data were obtained on test specimens with an end to side surface ratio of 1:20 and will apply primarily to dipping treatments of short length materials, e.g. window frames, doors, tent stakes, board walks, pickets, pallets, boxes, crates, etc.

Certain of the results should prove useful in increasing the effectiveness of dip treatments or in understanding their limitations.

The lack of appreciable penetration in specimens at the thirty-five and fifty per cent moisture contents demonstrated the inadequacy of the dip treatment of wood at moisture contents above the fiber saturation point.

Among specimens at moisture contents below the fiber saturation point only longitudinal penetration showed marked variations with the factors under study. This agreed with Schulze and Theden (1951) who reported no change in transverse absorption and penetration as a result of changes in moisture content below the fiber saturation point. The correlation of absorption and *longitudinal* penetration ($r = 0.752$) suggested that the highly significant differences among absorption data could probably be attributed almost entirely to *longitudinal* absorption. Therefore the effect of moisture content and specific gravity on absorption will depend upon the proportion of end grain present. This makes slope of grain and end to side grain ratios important dip treatment variables.

In wood products with a high proportion of side to end grain transverse penetration will be critical, and the slight radial and tangential penetrations obtained may be limiting factors in the utility of dip treatment.

Over half the total absorption occurred within fifteen seconds of immersion and thereafter the rate of absorption decreased with time. Rate of absorption also decreased with increased moisture content. Therefore the advantage gained by increasing the immersion period will decrease with increased moisture content of wood.

An increase in the drying temperature of wood effected an increase in absorption. Kiln-dried lumber can be expected to absorb more preservative than air-seasoned lumber.

Absorption varied inversely with the specific gravity of the wood. Increased absorptions and penetrations can be expected in low specific gravity ponderosa pine sapwood. However, these differences are of small magnitude and are believed to be of little practical significance. It is interesting to observe that the effect of specific gravity on absorption varies with moisture content. At moisture contents above the fiber saturation point specific gravity has less effect on absorption than it has at lower moisture contents.

Occasional specimens demonstrated anomalous absorption and penetration. This variation suggested that some factor(s), other than those being studied, might affect the amount of absorption.

Of fundamental interest are the mechanisms involved in the flow of liquids into or through wood. These mechanisms are complicated and not fully understood to date. The non-polar nature of the solvent limited movement of the preservative solutions to the gross capillary system of the wood. The initial absorption of the solutions may be explained in part by capillary forces causing flooding of the exposed cell lumens on the wood surfaces. This initial absorption was significantly reduced only in specimens at moisture contents above the fiber saturation point where accumulation of free water in the cell lumens would reduce available volume.

In test specimens at moisture contents below the fiber saturation point and beyond the period of initial absorption, increased moisture content caused significant decreases in rate of absorption. In addition, this increased moisture content caused a change in the effect of specific gravity. The mechanisms for these phenomena are unknown to the writers. The decreased permeability of wood with increased moisture content (due to reduction in size of pit membrane pores and checks) is a possible partial explanation. A change in the solution-wood interfacial tensions with increased wood moisture content might also be a partial explanation.

The combined effect of time of immersion and specific gravity and moisture content of wood on absorption and penetration of one of the preservative solutions is shown in Figure 4.

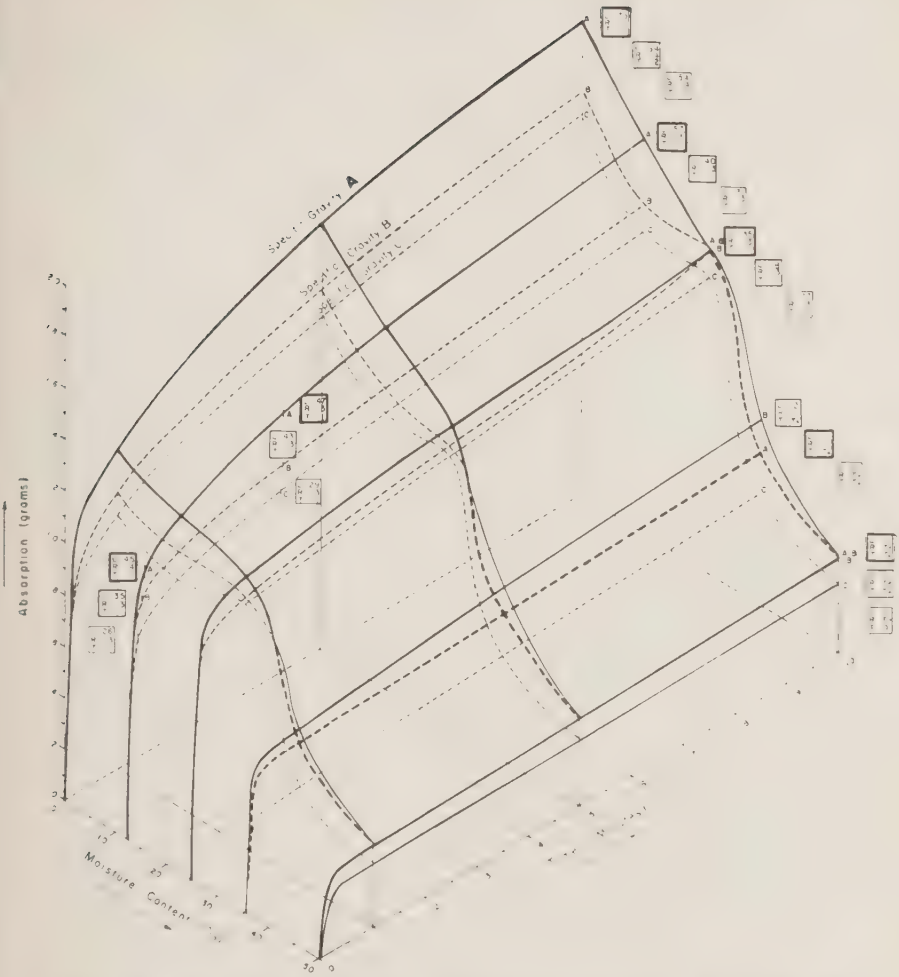

FIG. 4.—The absorption of a five per cent pentachlorophenol solution by ponderosa pine test specimens in a dip treatment. Specific gravity ranges A (0.33-0.39), B (0.40-0.44), and C (0.45-0.61) are represented by three planes delineated by different weight lines. Values of longitudinal (L), radial (R), and tangential (T) penetration (in millimeters) of the toxicant are noted in boxes adjacent to the corresponding points on the curves. "T" indicates trace penetration.

CONCLUSIONS

Moisture content of wood has a marked effect on the absorption and penetration of oil-soluble preservatives applied by dip treatment. These data suggest that wood at moisture contents above the fiber saturation point cannot be effectively treated by dipping. Below the fiber saturation point, transverse penetrations are not measurably affected by changes in moisture content; however, longitudinal penetration and total absorption are significantly affected. Initial absorption does not vary appreciably with changes in moisture contents below the fiber saturation point but does decrease at moisture contents above the fiber saturation point. In oven-dry wood the initial absorption accounts for approximately half the total absorption, and this proportion increases with an increase in moisture content.

An increase in the specific gravity of wood causes a slight decrease in absorption and penetration. This effect decreases with an increase in moisture content.

No important differences were apparent in the absorption and penetration characteristics of the several preservative solutions.

Maximum penetration occurred along the grain, while the least penetration occurred in the tangential direction. In the absence of appreciable exposed end grain low transverse penetrations may limit the utility of dip treatments.

Certain colorific organic indicators proved useful for penetration determinations.

These conclusions apply only to the materials and conditions tested.

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APPENDIX

Formulation of Standard Preservative Solutions[1]

5 per cent pentachlorophenol[2]

157.5 gm. Dowicide 7 (obtained from The Dow Chemical Co., Midland, Mich.)
300.0 gm. Pentalyn H[3]
48.0 gm. Fisher NF Paraffin (refined)
3207.0 ml. Solvasol No. 5[4]

2 per cent metallic copper (copper naphthenate)

759 gm. of 7.9 per cent copper naphthenate concentrate (obtained from Nuodex Products Inc., Elizabeth, N. J.)
300 gm. Pentalyn H
48 gm. Fisher NF Paraffin (refined)
2432 ml. Solvasol No. 5

1.1 per cent copper-8-quinolinolate

300 gm. Cunilate No. 2171—10 per cent concentrate (obtained from Scientific Oil Inc., Chicago, Ill.)
300 gm. Pentalyn H
48 gm. Fisher NF Paraffin (refined)
2670 ml. Solvasol No. 5

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[1] All per cent concentrations are calculated on a weight basis.

[2] 50.0 ml. pine oil added to solution; a 95 per cent assay of chlorinated phenols was used in calculating amount of chemical.

[3] Pentalyn H is the pentaerythritol ester of hydrogenated rosin and a product of Hercules Powder Co.

[4] Solvasol No. 5 is a Socony-Vacuum Oil Company, Inc., product with approximately 20 per cent aromatic content and a flash point of 103°F.

7.5 per cent rosin amine D pentachlorophenate

500 gm. SOCCI 2629—50 per cent concentrate (obtained from Scientific Oil Inc., Chicago, Ill.)
300 gm. Pentalyn H
48 gm. Fisher NF Paraffin (refined)
2835 ml. Solvasol No. 5

5 per cent zinc alkyl sulphonate

300 gm. N-136—50 per cent concentrate (obtained from Nuodex Products Inc., Elizabeth, N. J.)
300 gm. Pentalyn H
48 gm. Fisher NF Paraffin (refined)
3020 ml. Solvasol No. 5